

# **METAL MATRIX COMPOSITE**

## **PRODUCTION AND CHARACTERISATION OF ALUMINIUM-FLY ASH COMPOSITE USING STIR CASTING METHOD**

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE  
REQUIREMENTS FOR THE DEGREE OF

**Bachelor of Technology**

**In**

**Metallurgical & Materials Engineering**

**By**

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**Department of Metallurgical & Materials Engineering**

**National Institute of Technology**

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**2008**



## **National Institute of Technology Rourkela**

### **CERTIFICATE**

This is to certify that the thesis entitled, “Production and Characterisation of Aluminium-Fly ash composite using Stir Casting Method” submitted by Sudipt Kumar (10404002) and J.Ananda Theerthan (10304037) in partial fulfillment of the requirements for the award of Bachelor of Technology Degree in Metallurgical & Materials Engineering at the National Institute Of Technology, Rourkela (Deemed University) is an authentic work carried out by them under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

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## CONTENTS

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	<b>PAGE Number</b>
<b>Abstract</b> .....	9
<b>CHAPTER-1: INTRODUCTION</b>	10
1.1 INTRODUCTION	11-13
<b>CHAPTER-2: LITERATURE SURVEY</b>	14
2.1 COMPOSITE	15-16
2.2 CLASSIFICATION OF COMPOSITES	16-19
2.3 RULE OF MIXTURES	19-21
2.4 METAL MATRIX COMPOSITES	21-25
2.5 STIR CASTING METHOD	25-27
2.6 STRENGTHENING MECHANISM OF COMPOSITES	27-29
2.7 FLY ASH	29-31
2.8 CLASSIFICATION OF FLY ASH	31-33
2.9 WHY FLY ASH	33-34
2.10 CHEMICAL REACTION BETWEEN AI AND FLY ASH	34-35
2.11 INTERFACIAL PARAMETERS	35-40
<b>CHAPTER-3: EXPERIMENTAL WORK</b>	41
3.1 EXPERIMENTAL PROCEDURE	42-43

<b>CHAPTER-4: RESULTS AND DISCUSSION</b>	<b>44</b>
4.1 CHEMICAL ANALYSIS OF FLY ASH	45
4.2 PARTICLE SIZE ANALYSIS OF FLY ASH	45-46
4.3 DENSITY AND HARDNESS MEASUREMENT	46-47
4.4 WEAR BEHAVIOUR	47-49
4.5 SEM ANALYSIS	49-50
4.6 EDS MICROANALYSIS	51-52
<b>CHAPTER-5: CONCLUSION</b>	<b>53-54</b>
<b>CHAPTER-6: REFERENCES</b>	<b>55-57</b>

## LIST OF FIGURES

FIGURE NUMBER	NAME OF THE FIGURE	PAGE NUMBER
2.5	STIR CASTING	27
2.11 (a)	Cu-Sic INTERFACE	35
2.11(b)	LIQUID DROP ON THE SOLID SURFACE	37
4.2	PARTICLE SIZE ANALYSIS	45
4.4 (a)	WEAR Vs TIME	47
4.4 (b)	COF Vs TIME	48
4.5 (a)	SEM MICROSTRUCTURE OF AS CAST Al	49
4.5 (b)	SEM MICROSTRUCTURE OF AS Al-10% FLY ASH COMPOSITE	50
4.6 (a)	EDS MICROANALYSIS FOR AS CAST Al	51
4.6 (b)	EDS MICROANALYSIS FOR Al-10% FLY ASH COMPOSITE	52

**LIST OF TABLES**

<b>TABLE NUBER</b>	<b>NAME</b>	<b>PAGE NUMBER</b>
2.8	CHEMICAL COMPOSITION OF FLY ASH	31
4.1	CHEMICAL ANALYSIS OF FLY ASH	45
4.3	DENSITY AND HARDNESS MEASUREMENT	46
4.6 (a)	EDS MICROANALYSIS FOR AS CAST Al	51
4.6 (b)	EDS MICROANALYSIS FOR Al-10% FLY ASH COMPOSITE	52



## ABSTRACT

Metal matrix composites (MMCs) possess significantly improved properties including high specific strength; specific modulus, damping capacity and good wear resistance compared to unreinforced alloys. There has been an increasing interest in composites containing low density and low cost reinforcements. Among various discontinuous dispersoids used, fly ash is one of the most inexpensive and low density reinforcement available in large quantities as solid waste by-product during combustion of coal in thermal power plants. Hence, composites with fly ash as reinforcement are likely to overcome the cost barrier for wide spread applications in automotive and small engine applications. It is therefore expected that the incorporation of fly ash particles in aluminium alloy will promote yet another use of this low-cost waste by-product and, at the same time, has the potential for conserving energy intensive aluminium and thereby, reducing the cost of aluminium products. Now a days the particulate reinforced aluminium matrix composite are gaining importance because of their low cost with advantages like isotropic properties and the possibility of secondary processing facilitating fabrication of secondary components. The present investigation has been focused on the utilization of abundantly available industrial waste fly-ash in useful manner by dispersing it into aluminium to produce composites by stir casting method.

**Key words:** particulate composites, industrial waste, applied load and sliding velocity

# CHAPTER 1

## INTRODUCTION

## **1.1 INTRODUCTION**

Conventional monolithic materials have limitations in achieving good combination of strength, stiffness, toughness and density. To overcome these shortcomings and to meet the ever increasing demand of modern day technology, composites are most promising materials of recent interest. Metal matrix composites (MMCs) possess significantly improved properties including high specific strength; specific modulus, damping capacity and good wear resistance compared to unreinforced alloys. There has been an increasing interest in composites containing low density and low cost reinforcements. Among various discontinuous dispersoids used, fly ash is one of the most inexpensive and low density reinforcement available in large quantities as solid waste by-product during combustion of coal in thermal power plants. Hence, composites with fly ash as reinforcement are likely to overcome the cost barrier for wide spread applications in automotive and small engine applications. It is therefore expected that the incorporation of fly ash particles in aluminium alloy will promote yet another use of this low-cost waste by-product and, at the same time, has the potential for conserving energy intensive aluminium and thereby, reducing the cost of aluminium products [1-3].

Now a days the particulate reinforced aluminium matrix composite are gaining importance because of their low cost with advantages like isotropic properties and the possibility of secondary processing facilitating fabrication of secondary components. Cast aluminium matrix particle reinforced composites have higher specific strength, specific modulus and good wear

resistance as compared to unreinforced alloys [4-6]. While investigating the opportunity of using fly-ash as reinforcing element in the aluminium melt, R.Q.Guo and P.K.Rohatagi [7-8] observed that the high electrical resistivity, low thermal conductivity and low density of fly-ash may be helpful for making a light weight insulating composites. The particulate composite can be prepared by injecting the reinforcing particles into liquid matrix through liquid metallurgy route by casting [9-10]. Casting route is preferred as it is less expensive and amenable to mass production. Among the entire liquid state production routes, stir casting is the simplest and cheapest one. The only problem associated with this process is the non uniform distribution of the particulate due to poor wet ability and gravity regulated segregation.

Mechanical properties of composites are affected by the size, shape and volume fraction of the reinforcement, matrix material and reaction at the interface. These aspects have been discussed by many researchers. Rohatgi [1] reports that with the increase in volume percentages of fly ash, hardness value increases in Al-fly ash (precipitator type) composites. He also reports that the tensile elastic modulus of the ash alloy increases with increase in volume percent (3–10) of fly ash. Aghajanian et al. [11] have studied the  $\text{Al}_2\text{O}_3$  particle reinforced Al MMCs, with varying particulate volume percentages (25, 36, 46, 52 and 56) and report improvement in elastic modulus, tensile strength, compressive strength and fracture properties with an increase in the reinforcement content. The interface between the matrix and reinforcement plays a critical role in determining the properties of MMCs. Stiffening and strengthening rely on load transfer across the interface. Toughness is influenced by the crack deflection at the interface and ductility is affected by the relaxation of peak stress near the interface [12-14].

Extensive studies on the tribological characteristics of Al MMCs containing reinforcements such as SiC and Al<sub>2</sub>O<sub>3</sub> is available in the literatures [15-18]. However, reports on friction and wear characteristics of fly ash reinforced AMCs are very limited. Rohatgi has reported that the addition of fly ash particles to the aluminium alloy significantly increases its abrasive wear resistance. He attributed the improvement in wear resistance to the hard aluminosilicate constituent present in fly ash particles.

In the present work, fly-ash which mainly consists of refractory oxides like silica, alumina, and iron oxides is used as reinforcing phase. Composite was produced with 10% fly-ash as reinforcing phase. Commercially pure aluminium was also melted and casted. Then particle size and chemical composition analysis for fly-ash was done. Mechanical, physical and wear properties of the composite were evaluated and compared with the commercially pure aluminium. Moreover, the composite was characterized with the help SEM and EDS.

# CHAPTER 2

## LITERATURE SURVEY

## 2.1 COMPOSITE

**Composite material** is a material composed of two or more distinct phases (matrix phase and reinforcing phase) and having bulk properties significantly different from those of any of the constituents. Many of common materials (metals, alloys, doped ceramics and polymers mixed with additives) also have a small amount of dispersed phases in their structures, however they are not considered as composite materials since their properties are similar to those of their base constituents (physical property of steel are similar to those of pure iron) . Favorable properties of composites materials are high stiffness and high strength, low density, high temperature stability, high electrical and thermal conductivity, adjustable coefficient of thermal expansion, corrosion resistance, improved wear resistance etc.

### MATRIX PHASE

1. The primary phase, having a continuous character,
2. Usually more ductile and less hard phase,
3. Holds the reinforcing phase and shares a load with it.

### REINFORCING PHASE

1. Second phase (or phases) is imbedded in the matrix in a discontinuous form,
2. Usually stronger than the matrix, therefore it is sometimes called reinforcing phase.

**Composites as engineering materials normally refer to the material with the following characteristics:**

1. These are artificially made (thus, excluding natural material such as wood).
2. These consist of at least two different species with a well defined interface.
3. Their properties are influenced by the volume percentage of ingredients.
4. These have at least one property not possessed by the individual constituents.

**Performance of Composite depends on:**

1. Properties of matrix and reinforcement,
2. Size and distribution of constituents,
3. Shape of constituents,
4. Nature of interface between constituents.

## **2.2 CLASSIFICATION OF COMPOSITES**

Composite materials are classified

- a. On the basis of matrix material,
- b. On the basis of filler material.



**(a) On the basis of Matrix:****1. Metal Matrix Composites (MMC)**

Metal Matrix Composites are composed of a metallic matrix (aluminium, magnesium, iron, cobalt, copper) and a dispersed ceramic (oxides, carbides) or metallic (lead, tungsten, molybdenum) phase.

**2. Ceramic Matrix Composites (CMC)**

Ceramic Matrix Composites are composed of a ceramic matrix and imbedded fibers of other ceramic material (dispersed phase).

**3. Polymer Matrix Composites (PMC)**

Polymer Matrix Composites are composed of a matrix from thermoset (Unsaturated polyester (UP), Epoxy) or thermoplastic (PVC, Nylon, Polysterene) and embedded glass, carbon, steel or Kevlar fibers (dispersed phase).

**(b) On the basis of Material Structure:****1. Particulate Composites**

Particulate Composites consist of a matrix reinforced by a dispersed phase in form of particles.

1. **Composites with random orientation of particles.**
2. **Composites with preferred orientation of particles.** Dispersed phase of these materials consists of two-dimensional flat platelets (flakes), laid parallel to each other.

**2. Fibrous Composites**

1. **Short-fiber reinforced composites.** Short-fiber reinforced composites consist of a matrix reinforced by a dispersed phase in form of discontinuous fibers (length < 100\*diameter).
1. **Composites with random orientation of fibers.**
2. **Composites with preferred orientation of fibers.**

2. **Long-fiber reinforced composites.** Long-fiber reinforced composites consist of a matrix reinforced by a dispersed phase in form of continuous fibers.

1. **Unidirectional orientation of fibers.**
2. **Bidirectional orientation of fibers (woven).**

### 3. Laminate Composites

When a fiber reinforced composite consists of several layers with different fiber orientations, it is called **multilayer (angle-ply) composite**.

## 2.3 RULE OF MIXTURES

**Rule of Mixtures** is a method of approach to approximate estimation of composite material properties, based on an assumption that a composite property is the volume weighed average of the phases (matrix and dispersed phase) properties.

According to Rule of Mixtures properties of composite materials are estimated as follows:

### Density

$$d_c = d_m * V_m + d_f * V_f$$

Where

$d_c, d_m, d_f$  – densities of the composite, matrix and dispersed phase respectively;

$V_m, V_f$  – volume fraction of the matrix and dispersed phase respectively.

### Coefficient of Thermal Expansion

- *Coefficient of Thermal Expansion (CTE) in longitudinal direction (along the fibers)*

$$\alpha_{cl} = (\alpha_m * E_m * V_m + \alpha_f * E_f * V_f) / (E_m * V_m + E_f * V_f)$$

Where

$\alpha_{cl}, \alpha_m, \alpha_f$  – CTE of composite in longitudinal direction, matrix and dispersed phase (fiber) respectively;

$E_m, E_f$  – modulus of elasticity of matrix and dispersed phase (fiber) respectively.

- *Coefficient of Thermal Expansion (CTE) in transverse direction (perpendicular to the fibers)*

$$\alpha_{ct} = (1 + P_m) \alpha_m * V_m + \alpha_f * V_f$$

Where

$P_m$  – Poisson ratio of matrix.

**Poisson's ratio** is the ratio of transverse contraction strain to longitudinal extension strain in the direction of applied force.

### Modulus of Elasticity

- *Modulus of Elasticity in longitudinal direction ( $E_{cl}$ )*

$$E_{cl} = E_m * V_m + E_f * V_f$$

- *Modulus of Elasticity in transverse direction ( $E_{ct}$ )*

$$1/E_{ct} = V_m/E_m + V_f/E_f$$

## Tensile Strength

- *Tensile strength of long-fiber reinforced composite in longitudinal direction*

$$\sigma_c = \sigma_m * V_m + \sigma_f * V_f$$

Where

$\sigma_c$ ,  $\sigma_m$ ,  $\sigma_f$  – tensile strength of the composite, matrix and dispersed phase (fiber) respectively.

- *Tensile strength of short-fiber composite in longitudinal direction*

(fiber length is less than critical value  $L_c$ )

$$L_c = \sigma_f * d / \tau_c$$

Where

$d$  – diameter of the fiber;

$\tau_c$  – shear strength of the bond between the matrix and dispersed phase (fiber).

$$\sigma_c = \sigma_m * V_m + \sigma_f * V_f * (1 - L_c / 2L)$$

Where

$L$  – length of the fiber

- *Tensile strength of short-fiber composite in longitudinal direction*

(fiber length is greater than critical value  $L_c$ )

$$\sigma_c = \sigma_m * V_m + L * \tau_c * V_f / d$$

## 2.4 Metal Matrix Composites (MMCs)

Metal Matrix Composites are composed of a metallic matrix (Al, Mg, Fe, Cu etc) and a dispersed ceramic (oxide, carbides) or metallic phase (Pb, Mo, W etc). Ceramic reinforcement may be

silicon carbide, boron, alumina, silicon nitride, boron carbide, boron nitride etc. whereas Metallic Reinforcement may be tungsten, beryllium etc [19]. MMCs are used for Space Shuttle, commercial airliners, electronic substrates, bicycles, automobiles, golf clubs and a variety of other applications. From a material point of view, when compared to polymer matrix composites, the advantages of MMCs lie in their retention of strength and stiffness at elevated temperature, good abrasion and creep resistance properties [19]. Most MMCs are still in the development stage or the early stages of production and are not so widely established as polymer matrix composites. The biggest disadvantages of MMCs are their high costs of fabrication, which has placed limitations on their actual applications [20]. There are also advantages in some of the physical attributes of MMCs such as no significant moisture absorption properties, non-inflammability, low electrical and thermal conductivities and resistance to most radiations [21]. MMCs have existed for the past 30 years and a wide range of MMCs have been studied [19].

Compared to monolithic metals, MMCs have:

- Higher strength-to-density ratios
- Higher stiffness-to-density ratios
- Better fatigue resistance
- Better elevated temperature properties
  - Higher strength
  - Lower creep rate

- Lower coefficients of thermal expansion
- Better wear resistance

The advantages of MMCs over polymer matrix composites are:

- Higher temperature capability
- Fire resistance
- Higher transverse stiffness and strength
- No moisture absorption
- Higher electrical and thermal conductivities
- Better radiation resistance
- No out gassing
- Fabric ability of whisker and particulate-reinforced MMCs with conventional metalworking equipment.

Some of the disadvantages of MMCs compared to monolithic metals and polymer matrix composites are:

- Higher cost of some material systems
- Relatively immature technology
- Complex fabrication methods for fiber-reinforced systems (except for casting)
- Limited service experience

Numerous combinations of matrices and reinforcements have been tried since work on MMC began in the late 1950s. However, MMC technology is still in the early stages of development, and other important systems undoubtedly will emerge. Numerous metals have been used as matrices. The most important have been aluminum, titanium, magnesium, and copper alloys and superalloys.

The most important MMC systems are:

- **Aluminum matrix**

- Continuous fibers: boron, silicon carbide, alumina, graphite
- Discontinuous fibers: alumina, alumina-silica
- Whiskers: silicon carbide
- Particulates: silicon carbide, boron carbide

- **Magnesium matrix**

- Continuous fibers: graphite, alumina
- Whiskers: silicon carbide
- Particulates: silicon carbide, boron carbide

- **Titanium matrix**

- Continuous fibers: silicon carbide, coated boron



- Particulates: titanium carbide
- **Copper matrix**
  - Continuous fibers: graphite, silicon carbide
  - Wires: niobium-titanium, niobium-tin
  - Particulates: silicon carbide, boron carbide, titanium carbide.
- **Superalloy matrices**
  - Wires: tungsten

## 2.5 STIR CASTING METHOD OF FABRICATION OF MMCs

**Liquid state fabrication of Metal Matrix Composites** involves incorporation of dispersed phase into a molten matrix metal, followed by its Solidification.

In order to provide high level of mechanical properties of the composite, good interfacial bonding (wetting) between the dispersed phase and the liquid matrix should be obtained.

Wetting improvement may be achieved by coating the dispersed phase particles (fibers). Proper coating not only reduces interfacial energy, but also prevents chemical interaction between the dispersed phase and the matrix.

The simplest and the most cost effective method of liquid state fabrication is ***Stir Casting***.

### **Stir Casting**

**Stir Casting** is a liquid state method of composite materials fabrication, in which a dispersed phase (ceramic particles, short fibers) is mixed with a molten matrix metal by means of mechanical stirring.

The liquid composite material is then cast by conventional casting methods and may also be processed by conventional Metal forming technologies.

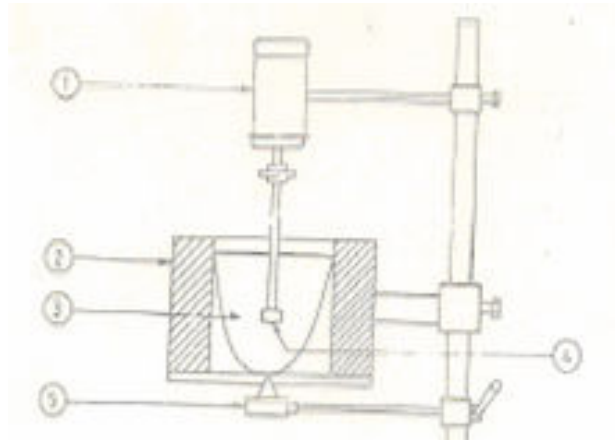
***Stir Casting is characterized by the following features:***

- Content of dispersed phase is limited (usually not more than 30 vol. %).
- Distribution of dispersed phase throughout the matrix is not perfectly homogeneous:
  1. There are local clouds (clusters) of the dispersed particles (fibers);
  2. There may be gravity segregation of the dispersed phase due to a difference in the densities of the dispersed and matrix phase.
- The technology is relatively simple and low cost.

Distribution of dispersed phase may be improved if the matrix is in semi-solid condition.

The method using stirring metal composite materials in semi-solid state is called **Rheocasting**.

High viscosity of the semi-solid matrix material enables better mixing of the dispersed phase.



**FIG 2.5 STIR CASTING**

1-Motor with stirring system,2-Heating Furnace,3-Crucible,4-Stirring blade,5-Plug.

## **2.6 STRENGTHENING MECHANISM OF COMPOSITES**

The strengthening mechanisms of the composites are different with different kind of reinforcing agent morphology such as fibres, particulate or dispersed type of reinforcing elements.

### **STRENGTHENING MECHANISM OF FIBRE REINFORCED COMPOSITE**

In such type of composite the reinforcing phase carries the bulk of the load and the matrix transfers the load to the reinforcing phase by the mechanism of shear. The high strength of the reinforcing phase restrict the free elongation of the matrix especially in its vicinity, whereas later is free to elongate at some distance away from the former.

This type of non uniform deformation of the matrix leads to a shear stress at the matrix reinforcement interface which results tensile stress at the reinforcing phase. Thus the stress is transferred to the reinforcing phase. The fibers either may be continuous or discontinuous in the matrix. In the former case the load is directly applied to the reinforcing phase and stress is constant over its entire length. In case of discontinuous fibers, the stress in the fibre increased from zero value at the end to a maximum value in the centre and thus average tensile strength developed is always less than those of continuous fibers. For the same when the fracture of the reinforcing phase, therefore the strength of the discontinuous fibre reinforced composite increases with increasing the length of the fibre and artifacts that of the continuous fibre reinforced one. Also the strength of the fibre reinforced composite will be maximum when the fibres are aligned in the direction of the applied stress i.e in the isostrain condition. So the strength of this kind of composite depends on the volume fraction of the reinforcing element present in the composite, which can be determined by the simple rule of mixtures.

## **DISPERSION STRENGTHENING MECHANISM OF STRENGTHENED COMPOSITE**

In the dispersion strengthened composite the second phase reinforcing agents are finely dispersed in the soft ductile matrix. The strong particles restrict the motion of dislocations and strengthen the matrix. Here the main reinforcing philosophy is by the strengthening of the matrix by the dislocation loop formation around the dispersed

particles. Thus the further movement of dislocations around the particles is difficult. Degree of strengthening depend upon the several factors like volume % of dispersed phase, degree of dispersion, size and shape of the dispersed phase, inter particle spacing etc. In this kind of composite the load is mainly carried out by the matrix materials.

### **STRENGTHENING MECHANISM OF PARTICULATE COMPOSITE**

In the particulate reinforced composite the size of the particulate is more than 1  $\mu\text{m}$ , so it strengthens the composite in two ways. First one is the particulate carry the load along with the matrix materials and another way is by formation of incoherent interface between the particles and the matrix. So a larger number of dislocations are generated at the interface, thus material gets strengthened. The degree of strengthening depends on the amount of particulate (volume fraction), distribution, size and shape of the particulate etc.

## **2.7 FLY ASH**

**Fly ash** is one of the residues generated in the combustion of coal. It is an industrial by-product recovered from the flue gas of coal burning electric power plants. Depending upon the source and makeup of the coal being burned, the components of the fly ash produced vary considerably, but all fly ash includes substantial amounts of silica (silicon dioxide,  $\text{SiO}_2$ ) (both

amorphous and crystalline) and lime (calcium oxide, CaO). In general, fly ash consists of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> as major constituents and oxides of Mg, Ca, Na, K etc. as minor constituent. Fly ash particles are mostly spherical in shape and range from less than 1 µm to 100 µm [22] with a specific surface area, typically between 250 and 600 m<sup>2</sup>/kg. The specific gravity of fly ash vary in the range of 0.6-2.8 gm/cc. Physical properties of fly ash mainly depend on the type of coal burned and the burning conditions. Class F fly ash is generally produced from burning high rank (containing high carbon content) coals such as anthracite and bituminous coals, whereas, Class C fly ash is produced from low rank coals. Fly ash particles are classified into two types, precipitator and cenosphere. Generally, the solid spherical particles of fly ash are called precipitator fly ash and the hollow particles of fly ash with density less than 1.0 g cm<sup>-3</sup> are called cenosphere fly ash. One common type of fly ash is generally composed of the crystalline compounds such as quartz, mullite and hematite, glassy compound such as silica glass, and other oxides. The precipitator fly ash, which has a density in the range 2.0–2.5 g cm<sup>-3</sup> can improve various properties of selected matrix materials, including stiffness, strength, and wear resistance and reduce the density. Cenosphere fly ash, which consists of hollow fly ash particles, can be used for the synthesis of ultra-light composite materials due to its significantly low density, which is in the range 0.4–0.7 g cm<sup>-3</sup>, compared with the densities of metal matrices, which is in the range of 1.6–11.0 g cm<sup>-3</sup> [23]. Coal fly ash has many uses [24] including as a cement additive, in masonry blocks, as a concrete admixture, as a material in lightweight alloys, as a concrete aggregate, in flowable fill materials, in roadway/runway construction, in structural fill materials, as roofing granules, and in grouting. The largest application of fly ash is

in the cement and concrete industry, though, creative new uses for fly ash are being actively sought like use of fly ash for the fabrication of MMCs.

## 2.8 CHEMICAL COMPOSITION AND CLASSIFICATION OF FLY ASH

Component	Bituminous	Sub bituminous	Lignite
$\text{SiO}_2$ (%)	20-60	40-60	15-45
$\text{Al}_2\text{O}_3$ (%)	5-35	20-30	20-25
$\text{Fe}_2\text{O}_3$ (%)	10-40	4-10	4-15
$\text{CaO}$ (%)	1-12	5-30	15-40
$\text{LOI}$ (%)	0-15	0-3	0-5

**TABLE 2.8**

Fly ash material solidifies while suspended in the exhaust gases and is collected by electrostatic precipitators or filter bags. Since the particles solidify while suspended in the exhaust gases, fly ash particles are generally spherical in shape and range in size from 1  $\mu\text{m}$  to 100  $\mu\text{m}$ . They consist mostly of silicon dioxide ( $\text{SiO}_2$ ), which is present in two forms: amorphous, which is rounded and smooth, and crystalline, which is sharp, pointed and hazardous; aluminium oxide ( $\text{Al}_2\text{O}_3$ ) and iron oxide ( $\text{Fe}_2\text{O}_3$ ). Fly ashes are generally highly heterogeneous, consisting of a mixture of glassy particles with various identifiable crystalline phases such as quartz, mullite, and various iron oxides.

## **On the basis of Chemical Composition:**

Two classes of fly ash are defined by ASTM C618: Class F fly ash and Class C fly ash. The chief difference between these classes is the amount of calcium, silica, alumina, and iron content in the ash. The chemical properties of the fly ash are largely influenced by the chemical content of the coal burned (i.e., anthracite, bituminous, and lignite).

### **1. Class F fly ash**

The burning of harder, older anthracite and bituminous coal typically produces Class F fly ash. This fly ash is pozzolanic in nature, and contains less than 10% lime (CaO). Possessing pozzolanic properties, the glassy silica and alumina of Class F fly ash requires a cementing agent, such as Portland cement, quicklime, or hydrated lime, with the presence of water in order to react and produce cementitious compounds. Alternatively, the addition of a chemical activator such as sodium silicate (water glass) to a Class F ash can lead to the formation of a geopolymer.

### **2. Class C fly ash**

Fly ash produced from the burning of younger lignite or sub bituminous coal, in addition to having pozzolanic properties, also has some self-cementing properties. In the presence of water, Class C fly ash will harden and gain strength over time. Class C fly ash generally contains more than 20% lime (CaO). Unlike Class F, self-cementing Class C fly ash does not require an activator. Alkali and sulfate ( $\text{SO}_4$ ) contents are generally higher in Class C fly ashes.



## **On the basis of size, shape and structure:**

### **1. Precipitator fly ash**

It is spherical in nature, the spheres are solid and the density is in the range of  $2.0\text{--}2.5\text{ g cm}^{-3}$ .

### **2. Cenosphere fly ash**

It is also spherical in shape but these spheres are hollow, so the density of this kind of fly ash is very less as compared to the precipitator fly ash. Here density is less than  $1\text{ gm cm}^{-3}$  (0.3-0.6 gm/cc)

## **2.9 WHY FLY ASH?**

1. The preference to use fly ash as a filler or reinforcement in metal and polymer matrices is that fly ash is a byproduct of coal combustion, available in very large quantities (80 million tons per year) at very low costs since much of this is currently land filled. Currently, the use of manufactured glass microspheres has limited applications due mainly to their high cost of production. Therefore, the material costs of composites can be reduced significantly by incorporating fly ash into the matrices of polymers and metallic alloys. However, very little information is available on to aid in the design of composite materials, even though attempts have been made to incorporate fly ash in both polymer and metal matrices. Cenosphere fly ash

has a lower density than talc and calcium carbonate, but slightly higher than hollow glass [25]. The cost of cenosphere is likely to be much lower than hollow glass [25]. Cenosphere may turn out to be one of the lowest cost fillers in terms of the cost per volume.

2. The high electrical resistivity, low thermal conductivity and low density of fly-ash may be helpful for making a light weight insulating composites [7-8].

3. Fly ash as a filler in Al casting reduces cost, decreases density and increase hardness, stiffness, wear and abrasion resistance [23]. It also improves the machinability, damping capacity, coefficient of friction etc. which are needed in various industries like automotive etc.

4. As the production of Al is reduced by the utilization of fly ash. This reduces the generation of green house gases as they are produced during the bauxite processing and alumina reduction.

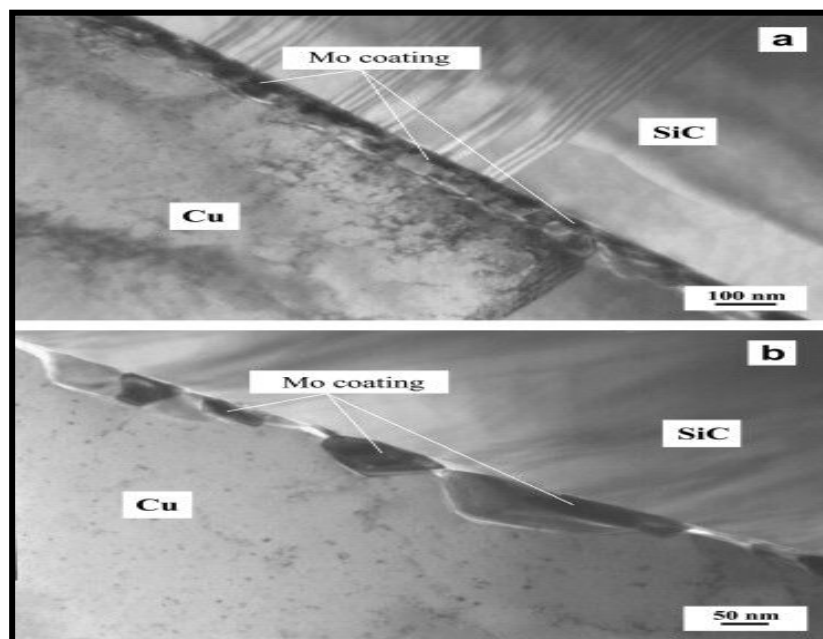
## **2.10 CHEMICAL REACTION BETWEEN Al AND FLY ASH**

The thermodynamic analysis indicates that there is possibility between the reaction of Al melt and the fly ash particles. The particles contain alumina, silica and iron oxide which during solidification process of Al fly ash composites or during holding such composites at temperature above 850<sup>0</sup> C, are likely to undergo chemical reactions, reported by P.K.Rohatagi and Guo. The experiments indicate that there is a progressive reduction between SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub> and mullite by Al

and formation of  $\text{Al}_2\text{O}_3$ , Fe and Si. The wall of cenosphere fly ash particles progressive disintegrates into discrete particles into the reaction progress.

## 2.11 INTERFACIAL PARAMETERS [19]

The desired properties of MMCs, namely strength, stiffness, fracture toughness, good creep and fatigue resistance are significantly influenced by the nature of the interface between reinforcement and the matrix.



**FIG 2.11 (a) Cu-SiC Interface**

**Criteria which are important in considering the interface that exists in a MMC material:**

**a. Adsorption and Wetting**

**b. Inter-diffusion**

**c. Chemical bonding**

**d. Mechanical adhesion**

**a. Adsorption and Wetting:**

Good wettability is needed to generate a strong interface that will allow transfer and distribution of load from the matrix to the dispersed phase, without premature

Thermodynamically reversible work needed to separate interface into its component parts:

$$W_a = \gamma_s + \gamma_L - \gamma_{SL} \dots\dots\dots (1)$$

$\gamma_s$  = Surface energy of solid

$\gamma_L$  = Surface energy of liquid

$\gamma_{SL}$  = Interfacial energy of solid-liquid phase

Young – Dupre's equation:

$$Y_S = Y_{SL} + Y_L \cos \phi \dots \dots \dots (2)$$

$\phi$  = contact angle

Combining (1) and (2)

$$W_a = Y_L (1 + \cos \phi)$$

Thus from a knowledge of  $\phi$  and  $Y_L$ , the work of adhesion can be found out.

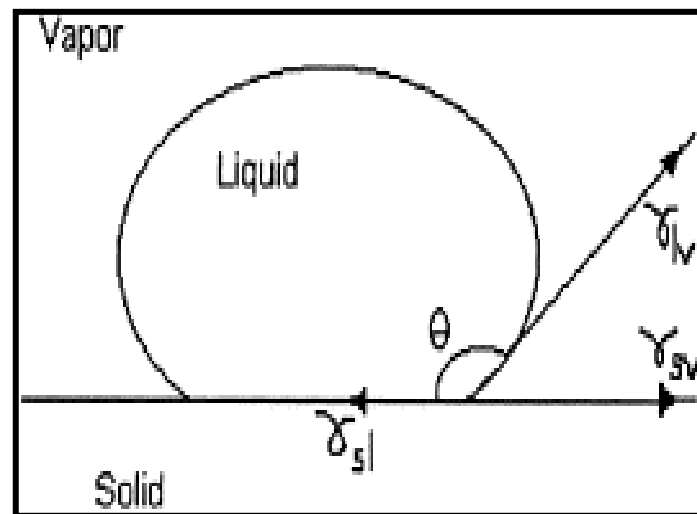


FIG 2.11 (b) LIQUID DROP ON THE SOLID SURFACE

1. For  $\theta=180^\circ$ , the drop is spherical with only point contact with the solid and no wetting takes place.
2. For  $\theta=0^\circ$ , the wetting is **PERFECT**.
3. For  $0^\circ < \theta < 180^\circ$ , the degree of wetting increases as  $\theta$  decreases.

**Adsorption** – It is a surface reaction which is dependent on concentration, temperature, and diffusivity. The greater the adsorption, the more the solute tends to lower the surface energy.

**b. Inter-diffusion-** Inter-diffusion plays only a minor role at low temperatures, but at elevated temperatures approaching the mp of the matrix, inter-diffusion and chemical reaction can result in the formation of brittle intermetallics which are detrimental to the mechanical properties of MMCs.

**c. Chemical bonding-**For strong chemical bonding between the reinforcement and matrix a controlled amount of chemical reaction at the interface is always desirable. However, too thick an interfacial zones adversely the mechanical properties of the composites and leads to premature failure.

Improvements in interfacial bonding in MMC are generally achieved by two means:

1. **Surface treatment of the reinforcement**-The reinforcement surfaces are coated with suitable materials to improve wettability and adhesion and also to prevent any adverse chemical reactions at elevated temperature.

Example - 1. Coatings of SiC on boron fibre for Al matrices.

2. B<sub>4</sub>C coating on boron fibres for Titanium matrices.

2. **Matrix Modification** - It is done by modifying the matrix alloy composition.

Example- 1. Alloying the Al matrix with Lithium promotes the wetting of polycrystalline Al<sub>2</sub>O<sub>3</sub> fibres. The Lithium is believed to react with alumina to form a lithium aluminate which is more readily wetted by aluminium.

*Alloying can minimize unwanted interfacial reactions.*

d. **Mechanical adhesion**-Mechanical bonding plays a major role in load transfer by shear. Two separate factors affect mechanical adhesion namely (1). Surface roughness, which control the amount of mechanical interlocking that can occur, and (2). The presence of residual stresses in the matrix as a result of fabrication.

Example- carbon and aluminium fibres possess small surface irregularities that affect the apparent fibre-matrix bond.

**POINTS TO REMEMBER:**

1. For strong chemical bonding between the reinforcement and matrix a controlled amount of chemical reaction at the interface is always desirable. However, too thick an interfacial zones adversely the mechanical properties of the composites and leads to premature failure.
2. Interfacial reactions are of concern as they can adversely affect the mechanical performance of an MMC. So, it is clear that, as a general rule, extensive interfacial reactions should be avoided if optimum mechanical performance is to be achieved with MMCs.



# CHAPTER 3

## EXPERIMENTAL WORK

### 3.1 EXPERIMENTAL PROCEDURE

First of all, 400 gm of commercially pure aluminium was melted in a resistance heated muffle furnace and casted in a clay graphite crucible. For this the melt temperature was raised to 993K and it was degassed by purging hexachloro ethane tablets. Then the aluminium-fly ash (10%) composite was prepared by stir casting route. For this we took 400 gm of commercially pure aluminium and 40 gm of fly ash. The fly ash particles were preheated to 373K for two hours to remove the moisture. Commercially pure aluminium was melted by raising its temperature to 993K and it was degassed by purging hexachloro ethane tablets. Then the melt was stirred using a mild steel stirrer. Fly-ash particles were added to the melt at the time of formation of vortex in the melt due to stirring. The melt temperature was maintained at 953K-993K during the addition of the particles. Then the melt was casted in a clay graphite crucible. The particle size analysis and chemical composition analysis was done for fly ash. The hardness testing and density measurement was carried out for both commercially pure Al and Al-10% fly ash composite. The hardness of the samples was determined by Brinell hardness testing machine with 500 kg load and 10 mm diameter steel ball indenter. The detention time for the hardness measurement was 30 seconds.

The wear characteristics of commercially pure Al and Al-10% fly ash composite were evaluated using wear testing machine. For this, cylindrical specimens of 1 cm diameter and 2.1 cm length were prepared from the cast aluminium and Al-10% fly ash composite. Test was performed at

68.68 N load and 500 rpm for 10 minutes. The SEM and EDS analysis was done for both the samples.

### **WORKS DONE:**

1. Commercially pure Al was melted and casted.
2. Al-10% fly ash composite was fabricated by stir casting method.
3. Chemical composition analysis was done for fly ash used.
4. Particle size analysis was done for fly ash used.
5. Density and hardness measurement was carried out for both commercially pure Al sample and Al-10% fly ash composite sample.
6. The wear characteristics of both commercially pure Al and Al-10% fly ash composite was evaluated and compared.
7. SEM analysis was done for both the samples.
8. EDS microanalysis was done for both the samples.

# CHAPTER 4

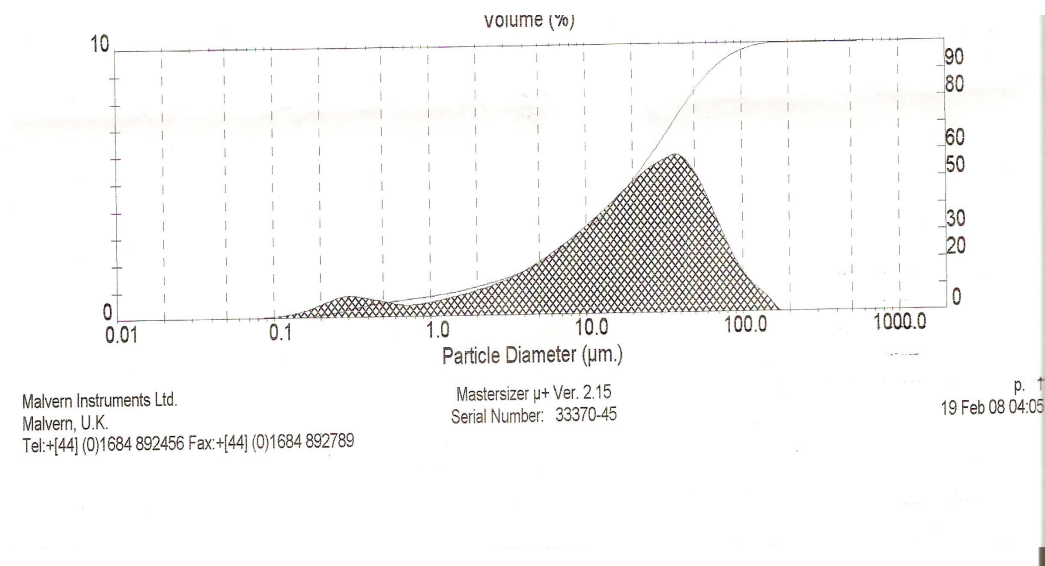
## RESULTS & DISCUSSION

## 4.1 CHEMICAL ANALYSIS OF FLY ASH

COMPOUNDS	PERCENTAGE (%)
SiO <sub>2</sub>	67.2
Al <sub>2</sub> O <sub>3</sub>	29.6
Fe <sub>2</sub> O <sub>3</sub>	0.1
CaO	1.4
MgO	1.7

**TABLE 4.1**

## 4.2 PARTICLE SIZE ANALYSIS OF FLY ASH



**FIG 4.2 PARTICLE SIZE ANALYSIS**

The size, density, type of reinforcing particles and its distribution have a pronounced effect on the properties of particulate composite. Size range of fly ash particles is reported in the above figure. The size range of the particles is very wide. The size ranges of the fly ash particles indicate that the composite prepared can be considered as dispersion strengthened as well as particle reinforced composite.

As is seen from the particle size distribution there are very fine particles as well as coarse ones (1-100  $\mu\text{m}$ ). Thus the strengthening of composite can be due to dispersion strengthening as well as due to particle reinforcement. Dispersion strengthening is due to the incorporation of very fine particles, which help to restrict the movement of dislocations, whereas in particle strengthening, load sharing is the mechanism.

### 4.3 DENSITY AND HARDNESS MEASUREMENT

SPECIMEN	DENSITY ( $\text{gm/cm}^3$ )	HARDNESS (BHN)
As Cast Al	3.398	16
Al-10% fly ash composite	2.807	18

**TABLE 4.3**

The above table shows that incorporation of fly ash particles in Aluminium matrix causes reasonable increase in hardness as well as reasonable decrease in density. The

strengthening of the composite can be due to dispersion strengthening as well as due to particle reinforcement.

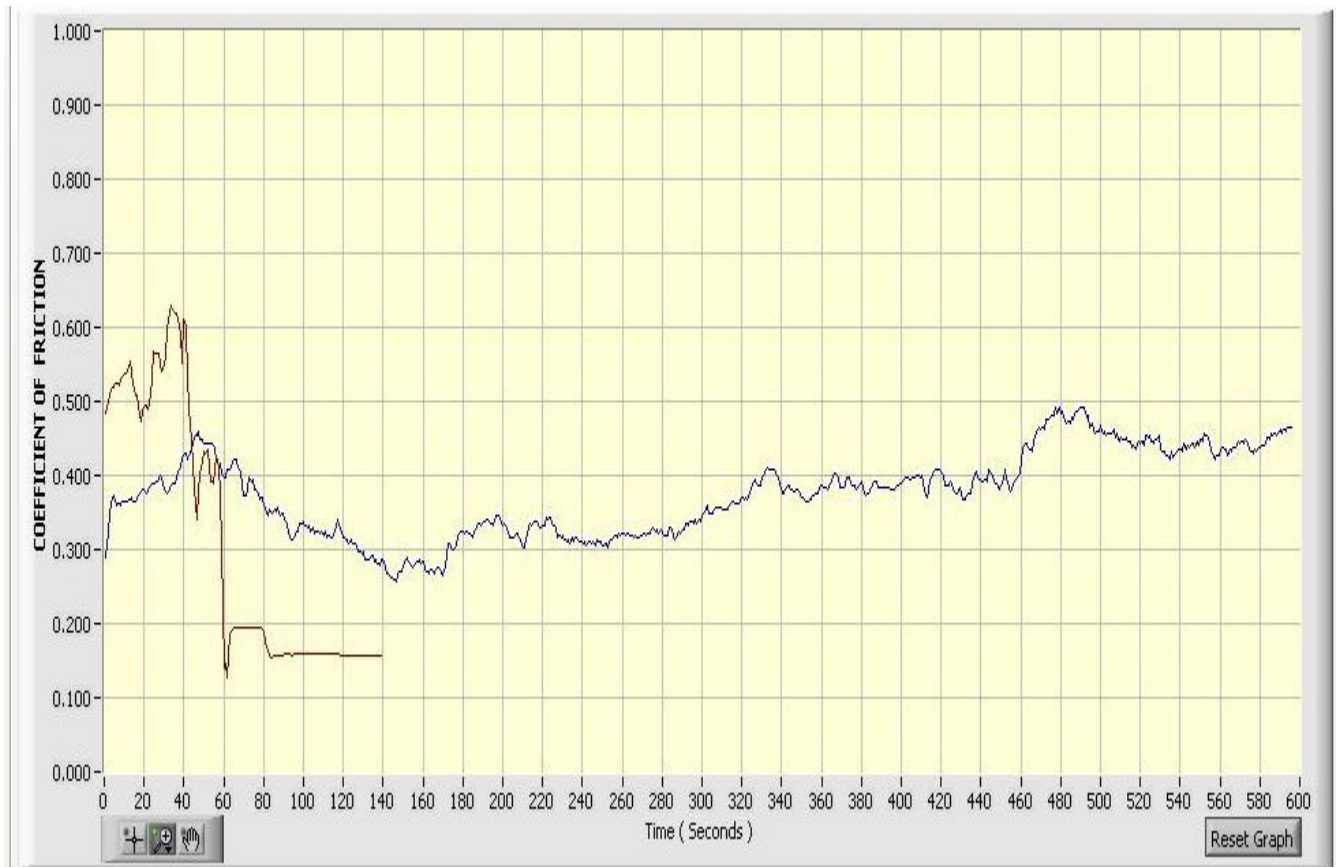
Thus, fly ash as filler in Al casting reduces cost, decreases density and increase hardness which are needed in various industries like automotive etc.

#### 4.4 WEAR BEHAVIOUR



FIG 4.4 (a) – Wear Vs Time

(RED-FOR Al, BLUE-FOR Al-10% FLY ASH)



**FIG 4.4 (b) COF Vs Time**

**(RED-FOR Al, BLUE-FOR Al-10% FLY ASH)**

**FOR FIG 4.4 (a)**

This figure clearly indicates that wear rate has improved significantly with the addition of fly-ash.

The addition of fly ash acts as a barrier to the movement of dislocations and thereby increases hardness of the composite. Thus addition of fly ash particles to the aluminium melt significantly increases its abrasive wear resistance. The improvement in wear resistance is due to the hard aluminosilicate constituent present in fly ash particles.



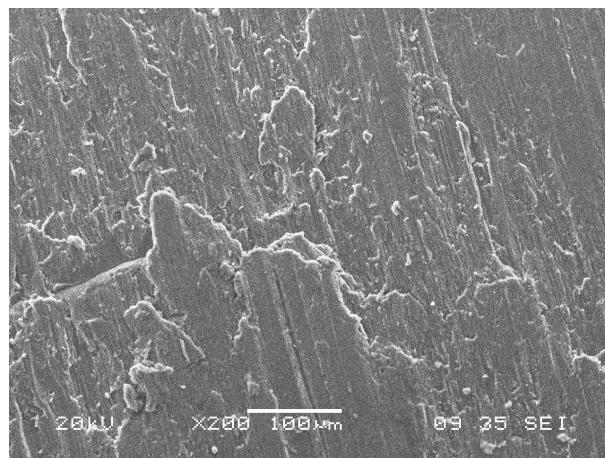
**FOR FIG 4.4 (b)**

This figure compares the typical friction coefficients of as cast Al and Al-10% fly ash composite. The Al-10% fly ash composite represents a lower friction coefficient than that of as cast Al. Thus friction coefficient decreases significantly with the incorporation of fly ash in Al melt.

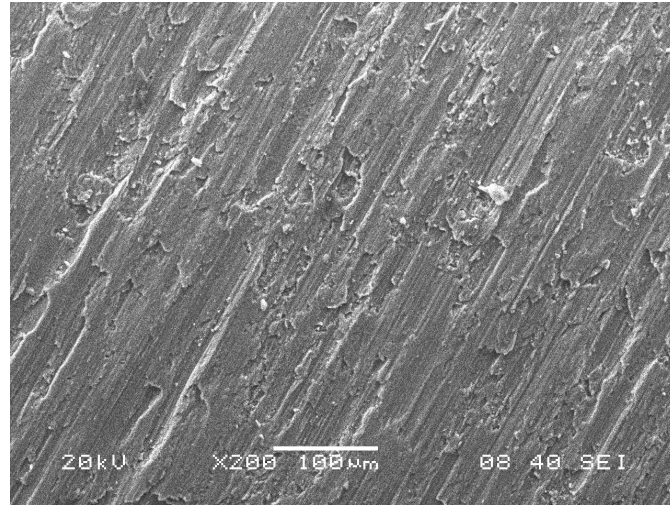
From the view of material, influencing factors on friction coefficient are mechanical properties of the matrix, hardness and chemical stability of the particles, and composition and strength of the interface. And interaction between these and tribological parameters (such as load and speed, environment and the properties of the counter faces materials) are responsible for the overall response.

**4.5 SEM ANALYSIS**

SEM photographs were taken to analyze the surfaces of as cast Al and Al-10% fly ash composite.



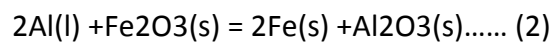
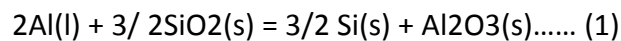
**FIG 4.5 (a) SEM microstructure of as cast Al**



**FIG 4.5 (b) SEM microstructure of Al-10% fly ash composite**

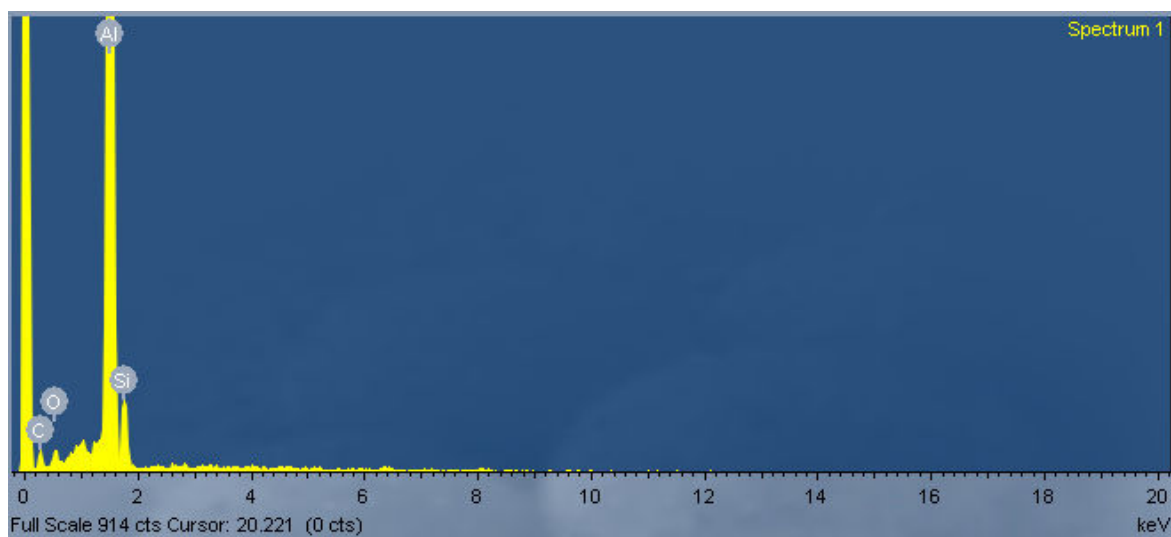
SEM analysis shows that more wear has occurred in case of as cast Al as compared to the Al-10% fly ash composite. This is due to the fact that wear is metallic wear in case of as cast Al where as wear is predominantly oxidative wear in case of Al-10% fly ash composite.

Thermodynamic analysis indicates that there is a possibility of chemical reaction between aluminium melt and fly ash particles. As these fly ash particles consist of alumina, silica and iron oxide, they are likely to undergo chemical reduction during their contact with the melt, as follows:



The elements (Si and Fe) formed by reduction reaction would alloy with the matrix. Gibbs free energy and the heats of reactions are highly exothermic in nature. As a result of this reaction (Eq. (1)) greater amount of eutectic silicon is seen in the composites.

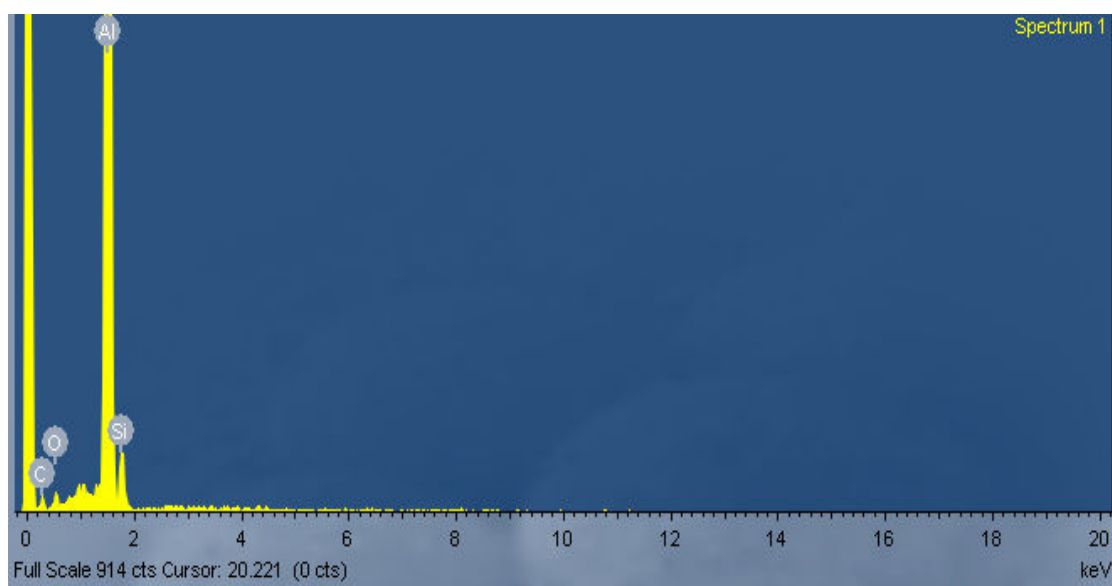
## 4.6 EDS (ENERGY DISPERSIVE SPECTROSCOPY) MICROANALYSIS



**FIG 4.6(a) EDS microanalysis for as cast Al**

Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corn.		Sigma	
C K	3.39	0.1817	20.14	2.64	35.46
O K	1.98	0.5478	3.89	1.00	5.14
Al K	80.78	1.2315	70.77	2.47	55.48
Si K	2.41	0.5007	5.20	0.46	3.91
Totals			100.00		

**TABLE 4.6 (a)**



**FIG 4.6(b) EDS microanalysis for Al-10% fly ash composite**

Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corn.		Sigma	
C K	2.79	0.1771	17.65	3.38	31.81
O K	2.10	0.5624	4.19	1.20	5.67
Al K	80.39	1.2386	72.73	3.14	58.34
Si K	2.39	0.4935	5.43	0.57	4.18
Totals			100.00		

**TABLE 4.6 (b)**

The increase in the amount of eutectic Si indicates the incorporation of fly ash in Al melt.

# CHAPTER 5

CONCLUSION

## CONCLUSION

1. From the study it is concluded that we can use fly ash for the production of composites and can turn industrial waste into industrial wealth. This can also solve the problem of storage and disposal of fly ash.
2. Fly ash upto 10% by weight can be successfully added to Al by stir casting route to produce composites.
3. The hardness of pure Al increased from 16 BHN to 18 BHN with addition of 10% fly ash. Moreover on addition of fly ash in Al melt, there was appreciable reduction of density from  $3.398 \text{ gm/cm}^3$  to  $2.807 \text{ gm/cm}^3$ .
4. Both the friction coefficients and the wear rates decreased significantly with the incorporation of fly ash in Al melt.
5. Strengthening of composite is due to dispersion strengthening and particle reinforcement .

# CHAPTER 6

## REFERENCES

## REFERENCES

- [1] P.K. Rohatgi, JOM 46 (11) (1994) 55–59.
- [2] T.P.D. Rajan, R.M. Pillai, B.C. Pai, K.G. Satyanarayana, P.K. Rohatgi, Proceedings of National Conference on: Recent Advances in Materials Processing (RAMP-2001), India, 2001, pp. 327–334.
- [3] P.K. Rohatgi, R.Q. Guo, P. Huang, S. Ray, Metall. Mater. Trans. A 28 (1997) 245–250.
- [4] Akbulut, M. Darman, F. Yilmaz, Wear 215 (1998) 170.
- [5] S. Skolianos, T.Z. Kattamis, Mater. Sci. Engg. A163 (1993) 107.
- [6] M.K. Surappa, S.C. Prasad, Wear 77 (1982) 295.
- [7] R.Q. Guo, P.K. Rohatgi, Fuel and energy Abstracts, (1997) 828.
- [8] R.Q. Guo, P.K. Rohatgi, Fuel and energy Abstracts, (1997) 157.
- [9] M.J. Koczak, M.K. Prem Kumar, JOM 45 (1993) 44.
- [10] P.C. Maity, P.N. Chakraborty, S.C. Panigrahi, Scripta Metall. Mater. 28 (1993) 549.
- [11] M.K. Aghajanian, R.A. Langensiepen, M.A. Rocazella, J.T. Leighton, C.A. Andersson, J. Mater. Sci. 28 (1993) 6683–6690.
- [12] T.W. Clyne, P.J. Withers, An Introduction to Metal Matrix Composites, Cambridge University Press, Cambridge, UK, 1993, pp. 166–217.
- [13] E.A. Feest, Composites 25 (1994) 75–86.
- [14] T.P.D. Rajan, R.M. Pillai, B.C. Bai, J. Mater. Sci. 33 (1998) 3491–3503.
- [15] L. Cao, Y. Wang, C.K. Yao, The wear properties of an SiC-whisker reinforced aluminium composite, Wear 140 (1990) 273–277.



- [16] B.N. Pramila Bai, B.S. Ramashesh, M.K. Surappa, Dry sliding wear of A356-Al-SiCp composites, *Wear* 157 (1992) 295–304.
- [17] Manish Narayan, M.K. Surappa, B.N. Pramila Bai, Dry sliding wear of Al alloy 2024–Al<sub>2</sub>O<sub>3</sub> particle metal matrix composites, *Wear* 181–183 (1995) 563–570.
- [18] A. Ravikiran, M.K. Surappa, Oscillations in coefficient of friction during dry sliding of A356 Al-30% wt. SiCp MMC against steel, *Scripta Mater.* 36 (1997) 95–98.
- [19] S.Bandyopadhyay, T.Das , and P.R.Munroe ,Metal Matrix Composites -The Light Yet Stronger Metals For Tomorrow, A Treaise On Cast materials, p-17-38.
- [20] T.W.Clyne, (2001), Metal Matrix Composites: Matrices and Processing, Encyclopedia of Materials : Science and Technology ,p- 8
- [21] Composite Materials: Engineering and Design by F.L.Matthews and R.D.rawlings, Chapman & Hall publication
- [22] Neville AM. Properties of concrete. 4th ed. NY, USA: John Wiley & sons, Inc.; 1996.
- [23] T. Matsunaga , J.K. Kim , S. Hardcastle and P.K. Rohatgi, Crystallinity and selected properties of fly ash particles, *Materials Science and Engineering A325* (2002) 333–343
- [24] 2004 Coal combustion product (CCP) production and use survey, American Coal Ash Association. ([http://www.acaa-usa.org/PDF/2004\\_CCP\\_Survey\(9-9-05\).pdf](http://www.acaa-usa.org/PDF/2004_CCP_Survey(9-9-05).pdf)).
- [25] E.C. Barber, Reduce Part Weight and Cast with Hollow Microspheres for Plastics, 3M Company, Technical Report, St. Paul, 1999.

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